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(54) Title: MILD COLD PEARLIZING CONCENTRATES

(57) Abstract

Novel cold pearlizing concentrates provide pearlescence to personal care and beauty care products, such as shampoos and beauty soaps, providing added luster and sheen. A mild, cold pearlizing concentrate is disclosed which comprises: a) from about 5 % to about 40 % of a pearlizing agent; b) from about 3 % to about 30 % of a nonionic surfactant; c) from about 2 % to about 20 % of an amphoteric surfactant; d) from 0 % to about 15 % of a glycol emulsifier; and e) the remainder water.

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Mild Cold Pearlizing Concentrates

Field of the Invention

The present invention relates generally to cold pearlizing concentrates used to impart pearl, iridescent sheen or glow, to beauty care and personal care products such as liquid soaps and shampoos.

Background of the Invention

Pearlescent additives, also known as pearlizing agents, are added to beauty and personal care products such as hair and skin care products to provide a pearly appearance to the products. Chemicals which are tiny (micron size) needles or platelets often exhibit this pearly appearance. Materials which exhibit this effect are ethylene glycol mono-and di-stearate, TiO₂ coated mica, bismuth oxychloride, and natural mother of pearl. Many organic materials exhibit this pearlescence provided they can be produced in an appropriate needle or platelet shape. Ethylene glycol distearate (EGDS) is the most commonly utilized pearlizing agent.

Obtaining good pearlescence requires obtaining the appropriate crystallization. Consistency in obtaining the appropriate size and type of crystal formed is difficult especially when utilizing ethylene glycol distearate or the like. The controlled formation of EGDS crystals of the proper size to give good pearlescence depends on two major steps of the crystallization process. The first step is the solubilization by addition of EGDS to the hot (above EGDS's melting point) beauty or personal care product, for example a shampoo. Good, efficient solubilization of the EGDS depends on being above the melting point, finely emulsifying the melted EGDS; and allowing enough mixing time for the solubilization to occur. As the shampoo composition, for example, is varied, it is generally necessary to insure that it can solubilize the EGDS at a temperature greater than EGDS's

5 melting point but then also precipitate it at a temperature less than the melting point. If a composition has too high a solubilizing capacity the EGDS may not crystallize out, may require too low of an outlet temperature (<100°F) to crystallize or may only crystallize slowly leading to crystals that are too large. If the solubilizing capacity is too low, only part of the EGDS will be solubilized and upon cooling the unsolubilized EGDS will freeze out
10 as large chunks.

In the second step, the precipitation or freezeout step, composition and the cooling process are important parameters. The composition should not have too high a solubilization ability as discussed. Generally this is controlled by adding water or other solubilization reducing ingredients immediately prior to freeze out. Several cooling processes are known in the art. Some methods nucleate crystals in only a portion of the composition and then mix these seed crystals back into the warmer composition. A single-pass method is better for controlling crystal size since it does not require remixing crystals into the uncooled composition. Many factors, such as flow rate, temperature and time, must be monitored and controlled to achieve appropriate crystal size. The formation of 15 crystals depends very much on the rate of cooling. An imperfect cooling will decrease the pearlescence and the heating and cooling may have to be repeated until a satisfactory 20 pearlescence is achieved.

Obviously achieving consistency in the crystallization process requires constant monitoring as well as attending to adjustments. The difficulty in controlling the process 25 translates to an inconsistency in the pearlescence of the compositions and expenditures in time and money.

A pearlizing concentrate which can be added to beauty and personal care compositions without a need for crystallization, with its requisite heating and cooling elements, can provide significant benefits. It can be added at room temperature saving 30 energy and equipment costs and offer a more consistent pearlescence since many of the sensitive parameters of a crystallization process have been removed.

Cocodiethanolamide has been used as a dispersing agent in manufacturing cold pearlizing concentrates since it is liquid at room temperature and thus does not require

5 a melting step; however, studies have raised concern that the diethanolamine, often present as a by-product of the cocodiethanolamide production, may form potentially carcinogenic nitrosamines. Thus, the use of cocodiethanolamide in personal hair care and skin products such as cosmetics, facial soaps and shampoos has been questioned from a health standpoint. It would certainly be advantageous then, to have a safe
10 pearlizing concentrate for use in beauty and personal care compositions such as shampoo, lipstick and conditioner products without the presence of the cocodiethanolamide as a component of the formulation.

It is an object of the present invention to prepare a novel mild cold pearlizing concentrate for use in beauty and personal care compositions such as shampoos, skin
15 creams, lipstick, hair colors and the like. More particularly, it is an object of the present invention to prepare a cold pearlizing concentrate that does not require the presence of the cocodiethanolamide with the potential health problem associated with the nitrosamines possibly formed by its diethanolamine by-product. It is an object to provide beauty and personal care product formulators with a convenient means of
20 providing pearlescence to their products. Also an object of the present invention is to provide mildness, ie, mild/nonirritating to the skin.

Unlike current pearlizing concentrates wherein anionic surfactants such as sodium laureth sulfate, are the main emulsifiers, which are not compatible with some cationic surfactants which are conditioners, it is an object to provide a pearlizing
25 concentrate which is compatible with any ionic surfactant. A further object is that the beauty and personal care products can be prepared without the need for the application of high temperatures and other requirements of standard crystallization.

Summary of the Invention

The present invention is a novel mild cold pearlizing concentrate for use in
30 beauty and personal care compositions such as: shampoos, conditioners, lipsticks, skin creams, lotions, bubble baths, liquid dishwashing products, liquid cleaners and the like. The cold pearlizing concentrate is a unique formulation of ingredients comprising: a pearlizing agent, a nonionic surfactant, an amphoteric surfactant, a glycol emulsifier

5 and water. The cold pearlizing concentrate provides a brilliant sheen and pearlescence when incorporated into personal care and beauty care products.

All percentages and ratios utilized herein are on a weight percent basis unless otherwise stated.

10 "Comprising," as used herein, means various components can be conjointly employed. Accordingly, the terms 'consisting essentially of' and 'consisting of' are embodied in the term comprising.

The term "cold" as utilized herein refers to the ability of the concentrate to be added without heating the beauty and personal care products.

Detailed Description of the Invention

15 A stable, mild free flowing cold pearlizing concentrate is prepared using i) a pearlizing agent, preferably a glycol stearate; ii) a nonionic surfactant; iii) an amphoteric surfactant emulsifier and stabilizer and iv) a glycol emulsifier and v) water; to obviate the use of cocodiethanolamide and provide excellent compatibility with any ionic surfactant.

20 The pearlizing agent comprises from about 5% to about 40%, preferably from about 10% to about 30% and most preferably from about 15% to about 25%, by weight based on the total weight of the concentrate.

25 The pearlizing agent can be selected from the group consisting of hydroxyl stearate, polyethylene glycol mono- and di-stearates, ethylene glycol mono- and distearates, stearic monoethanolamide, stearic monoethanolamide stearate and mixtures thereof. The preferred agents are polyethylene glycol mono- and distearates, and ethylene glycol mono- and di-stearates. The most preferred emulsifiers for use are: ethylene glycol mono- and di-stearates.

30 A second component of the concentrate is a nonionic surfactant. This surfactant can function as an emulsifier and stabilizer in the formulation. The term 'nonionic surfactant' as utilized herein encompasses mixtures of nonionic surfactants.

Examples of useful nonionic surfactants include condensates of ethylene oxide with a hydrophobic moiety which has an average hydrophilic lipophilic balance (HLB) between

5 about 8 to about 16, and more preferably, between about 10 and about 12.5. These surfactants include the condensation products of primary or secondary aliphatic alcohols having from about 8 to about 24 carbon atoms, in either straight or branched chain configuration, with from about 2 to about 40, and preferably between about 2 and about 9 moles of ethylene oxide per mole of alcohol.

10 In a preferred embodiment the aliphatic alcohol comprises between about 9 and about 18 carbon atoms and is ethoxylated with between about 3 and about 12 moles of ethylene oxide per mole of aliphatic alcohol. Especially preferred are the about 12 to about 15 carbon primary alcohol ethoxylates containing about 5 to about 9 moles of ethylene oxide per mole of alcohol. One such material is commercially sold under the trade name
15 Neodol 25-9 by Shell Chemical Company. Other commercial nonionic surfactants include Neodol 25-6.5 and Neodol 25-7 sold by Shell Chemical Company.

Other suitable nonionic surfactants include the condensation products of about 6 to about 12 carbon atom alkyl phenols with about 3 to about 30, and preferably between about 5 and 14 moles of ethylene oxide. Examples of such surfactants are sold under the
20 trade names Igepol CO 530, Igepol CO 630, Igepol CO720 and Igepol CO 730 by Rhone-Poulenc Inc. Still other suitable nonionic surfactants are described in U.S. Patent No. 3,976,586. To the extent necessary, this patent is expressly incorporated by reference.

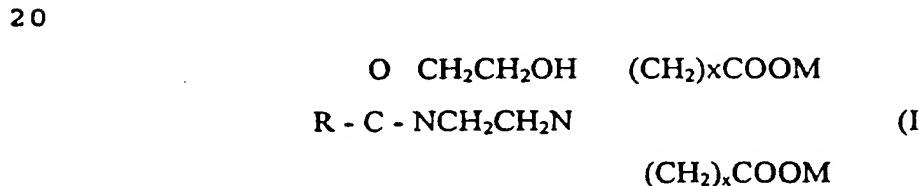
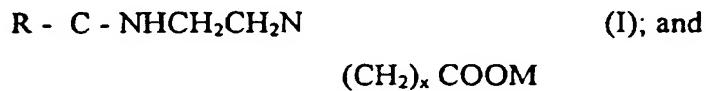
Most preferred for use are mixed linear alcohol ethoxylates such as Laureth-7 sold as Rhodasurf L-790 by Rhône-Poulenc Inc.

25 The nonionic surfactant is incorporated in the cold pearlizing concentrate in an amount of from about 3% to about 30%; preferably from about 8% to about 25% and most preferably from about 10% to 20%, based on the total weight of the concentrate.

An amphoteric surfactant comprises the third component of the present invention. The term "amphoteric surfactant" as utilized herein encompasses one or
30 more amphoteric surfactants such as mixtures of amphoteric surfactants. Preferably, amphoteric surfactants known as the betaines, their derivatives, and mixtures thereof are incorporated to provide an enhanced pearlizing effect.

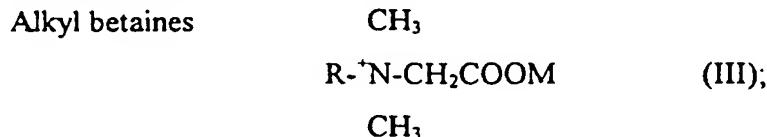
5 Examples of suitable amphoteric surfactants include the alkali metal, alkaline earth metal, ammonium or substituted ammonium salts of alkyl amphocarboxy glycinate and alkyl amphocarboxypropionate, alkyl amphodipropionate, alkyl amphodiacetate, alkyl amphoglycinate and alkyl amphopropionate wherein alkyl represents an alkyl group having 6 to 20 carbon atoms. Other suitable amphoteric 10 surfactants include alkyl iminopropionate, alkyl iminodipropionate and alkyl amphopropylsulfonates having between 12 and 18 carbon atoms; alkyl betaines and amidopropyl betaines and alkyl sulfonates and alkylamidopropylhydroxy sulfonates wherein alkyl represents an alkyl group having 6 to 20 carbon atoms.

15 Particularly useful amphoteric surfactants include both mono and dicarboxylates such as those of the formulae:

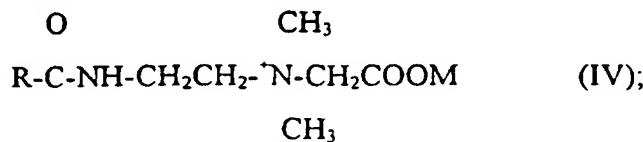


20 wherein R is an alkyl group of 6-20 carbon atoms, x is 1 or 2 and M is hydrogen or sodium. Mixtures of the above structures are particularly preferred.

25 Other formulae for the above amphoteric surfactants include the following:



30 Amidopropyl betaines

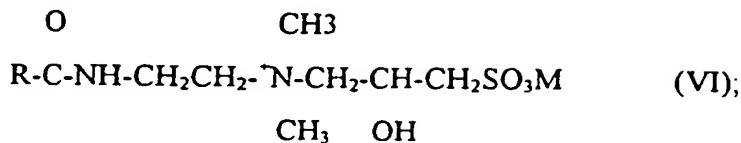


5 Alkyl sulfaines



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Alkyl amidopropylhydroxy sultaines



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where R is a alkyl group of 6-20 carbon atoms and M is potassium, sodium or a monovalent cation.

Of the above amphoteric surfactants, particularly preferred are the alkali salts of alkyl amphocarboxyglycinates and alkyl amphocarboxypropionates, alkyl amphodipropionates, alkyl amphodiacetates, alkyl amphoglycinates, alkyl amphopropyl sulfonates and alkyl amphopropionates wherein alkyl represents an alkyl group having 6 to 20 carbon atoms. Even more preferred are compounds wherein the alkyl group is derived from coconut oil or is a lauryl group, for example cocoamphodipropionate. Such cocoamphodipropionate surfactants are commercially sold under the trademarks Miranol C2M-SF CONC. and Miranol FBS by Rhone-Poulenc Inc.

Other commercially useful amphoteric surfactants include:

cocoamphoacetate (sold under the trademarks MIRANOL ULTRA C-32 and MIRAPON FA).

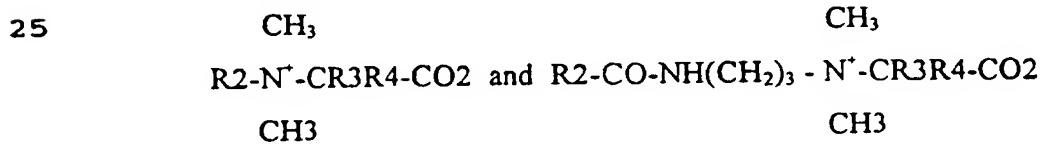
cocoamphopropionate (sold under the trademarks MIRANOL CMSF CONC. and MIRAPON FAS).

cocoamphodiacetate (sold under the trademarks MIRANOL C2M CONC. and MIRAPON FB).

5 lauroamphoacetate (sold under the trademarks MIRANOL HM CONC. and MIRAPON LA),
10 lauroamphodiacetate (sold under the trademarks MIRANOL H2M CONC. and MIRAPON LB),
lauroamphodipropionate (sold under the trademarks MIRANOL H2M-SF CONC. AND MIRAPON LBS),
15 lauroamphodiacetate obtained from a mixture of lauric and myristic acids (sold under the trademark MIRANOL BM CONC.), and
cocoamphopropyl sulfonate (sold under the trademark Miranol CS CONC.)
caproamphodiacetate (sold under the trademark MIRANOL S2M CONC.),
20 caproamphoacetate (sold under the trademark MIRANOL SM CONC.),
caproamphodipropionate (sold under the trademark MIRANOL S2M-SF CONC.), and
stearoamphoacetate (sold under the trademark MIRANOL DM).

The most preferred amphoteric surfactant for use is cocoamphoacetate. It can be present from 0% to 10% based on the total weight of the concentrate. Preferably, cocoamphoacetate will comprise from about 1% to about 7% and most preferably from about 2% to about 4% of the concentrate.

Also useful herein are the betaines and amidobetaines which are compounds of the general structure:



respectively wherein R₂ is C₈ - C₂₂ alkyl or alkenyl; R₃ is H or C₁ - C₄ alkyl; and R₄ is H or C₁ - C₄ alkyl.

30 The betaines useful herein include the high alkyl betaines such as cocodimethyl carboxymethyl betaine, lauryl dimethyl carboxymethyl betaine, lauryl dimethyl alpha-carboxy-ethyl betaine, cetyl dimethyl carboxymethyl betaine, lauryl bis-(2-hydroxy-

5 ethyl)carboxy methyl betaine, stearyl bis-(2-hydroxy-propyl)carboxymethyl betaine, oleyl dimethyl gamma-carboxypropyl betaine, and lauryl bis-(2-hydroxypropyl)alpha-carboxyethyl betaine. The sulfobetaines are also preferred and may be represented by cocodimethyl sulfopropyl betaine, stearyldimethyl sulfopropyl betaine, lauryl dimethyl sulfoethyl betaine, lauryl bis-(2-hydroxy-ethyl)sulfopropyl betaine and mixtures 10 thereof. A particularly preferred composition utilizes cocoamidopropyl betaine.

Most preferably, the amphoteric surfactant can be cocoamphoacetate and cocoamidopropyl betaine acting as amphoteric co-emulsifiers.

15 The amphoteric surfactant can be present from about 2% to about 20% weight percent based on the total weight of the pearlizing concentrate. Preferably, the amphoteric will comprise from about 4% to about 16%, most preferably from about 6% to about 10%, of the pearlizing concentrate.

20 The fourth component consists of a glycol emulsifier. Propylene glycol (1,2, and 1, 3) and other alcohols such as 1, 3 - butylene glycol, 2, 3 - butylene glycol, ethylene glycol and mixtures thereof are useful emulsifiers. The glycol emulsifier can be present from 0% to about 15%, preferably from about 1% to about 10% and most preferably from about 2% to about 5%.

25 For the fifth component, the remainder is water, preferably deionized. Generally, water is added in an amount of from about 20% to about 70%, preferably from about 30% to about 60%, and most preferably from about 40% to about 55% based on the total weight of the concentrate.

30 Non-essential optional components can be utilized in the concentrates of the present invention as a convenient means of incorporation into beauty and personal care products. Such conventional optional ingredients are well known to those skilled in the art, e.g., preservatives such as benzyl alcohol, methyl paraben, propyl paraben and imidazolidinyl urea; thickeners and viscosity modifiers such as block polymers of ethylene oxide and propylene oxide, e.g. Antarox F-88 (Rhone-Poulenc Inc.), sodium chloride, sodium sulfate, polyvinyl alcohol, and ethyl alcohol; pH adjusting agents such as citric acid, succinic acid, phosphoric acid, sodium hydroxide, sodium carbonate;

5 perfumes; dyes; and sequestering agents such as disodium ethylenediamine tetra-acetate. Such agents generally are used individually at levels of from 0% to about 2%, preferably from 0.01% to about 1.0% by weight of the concentrate.

10 The pH of the concentrate compositions is not critical and can be in the range of from about 2 to about 12, preferably from about 4 to about 10 and most preferably from about 6 to about 8. The pH can be adjusted using a buffer such as citric acid.

15 The order of addition to the mixing tank of the individual components of the concentrate is not critical nor is the reasonably elevated temperature; however, preferably the water and pearlizing agent are intimately blended at from about 50° to about 90°C, more preferably from about 70° to about 80°C with high agitation until the pearlizing agent is solubilized. The nonionic and amphoteric surfactants are then blended into the mix until the mixture is clear. The mixture is then allowed to cool to room temperature. Generally, the concentrate can be stored at a temperature of from about 0°C to about 45°C, preferably from about 15°C to about 35°C for at least one day and preferably two days in order to fully develop its pearlizing characteristics.

20 The cold pearlizing concentrate of the present invention can be specifically formulated into a wide variety of personal care and beauty care products. These products can be formulated by one skilled in the art utilizing conventional methods of production. The pearlizing concentrate imparts a high luster pearlescence and sheen to the products. Generally, the shampoos and soaps of the present invention can be made by merely mixing the beauty and/or personal care product together with the concentrate at room temperature.

25 The following examples are provided to better describe and define the concentrate compositions of the present invention. They are for illustrative purposes only, and it is realized that changes and variations may be made with respect to these compositions that are not shown below. Such changes that do not materially alter the compositions formulation or function are still considered to fall within the spirit and scope of the invention as recited by the claims that follow.

5

Example I

A cold pearlizing concentrate of the present invention is prepared as follows:

	Ethylene Glycol Distearate	20.0
	Laureth-7	15.0
10	Cocoamidopropyl Betaine	6.0
	Cocoamphoacetate	2.0
	Propylene Glycol	3.0
	Deionized Water	<u>54.0</u>
		100.00

15

Croda EGDS (Ethylene Glycol Distearate) is added to a mixture of water, followed by Laureth-7 (Rhodasurf L-790 sold by Rhône-Poulenc Inc), Cocoamidopropyl Betaine (Mirataine Bet-C-30 sold by Rhône-Poulenc Inc.) Cocoamphoacetate (Miranol Ultra C-32 sold by Rhône-Poulenc Inc.) and Propylene Glycol. The mixture is then heated to 80-85°C until the solution is clear (transparent). The solution is then allowed to cool to room temperature. The resulting concentrate has excellent pearlescence and can be utilized in a variety of personal and beauty care products.

Example II

A pearlescent mild conditioning shampoo is prepared as follows:

25	Sodium Laureth Sulfate (70%)	10.0
	Sodium Cocoamphoacetate (40%)	6.0
	Sodium Cocamidopropyl Betaine (36%)	4.0
	Pealizing Concentrate of Example I	2.0
	Guar Hydroxypropyl Trimonium Chloride	0.3
30	Citric Acid	q.s.
	NaCl	q.s.
	Frangrance/Dyes	q.s.
	Deionized Water	q.s.

- 5 The cold pearlizing concentrate of Example I is added to the premixed ingredients of the shampoo with agitation at room temperature. The resulting concentrate has excellent pearlescence and can be utilized to clean hair.

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What is Claim is:

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1. A mild, cold pearlizing concentrate comprising:
 - a) from about 5% to about 40% of a pearlizing agent;
 - b) from about 3% to about 30% of a nonionic surfactant;
 - c) from about 2% to about 20% of an amphoteric surfactant;
 - 10 d) from 0% to about 15% of a glycol emulsifier; and
 - e) the remainder water.
2. A composition according to claim 1 wherein the pearlizing agent is selected from the group consisting of hydroxy stearate, polyethylene glycol monostearate, polyethylene glycol distearate, ethylene glycol monostearate, 15 ethylene glycol distearate, stearic monoethanolamide, stearic monoethanolamide stearate, and mixtures thereof.
3. A composition according to claim 2 wherein said pearlizing agent is selected 20 from the group consisting of polyethylene glycol monostearate, polyethylene glycol distearate, ethylene glycol monostearate, ethylene glycol distearate and mixtures thereof.
4. A composition according to claim 2 comprising from about 10% to about 30% 25 pearlizing agent.
5. A composition according to claim 2 wherein said nonionic surfactant is selected from the group consisting of condensation products of primary and secondary 30 aliphatic alcohols having from about 8 to about 24 carbon atoms, in either straight or branched chain configuration, with from about 2 to about 40 moles of ethylene oxide per mole of alcohol; condensation products of about 6 to about 12 carbon atom alkyl phenols with about 3 to about 30 moles of ethylene oxide; and mixtures thereof.

5

6. A composition according to claim 5 wherein said nonionic surfactant comprises a condensation product of primary or secondary aliphatic alcohols having from about 9 to about 18 carbon atoms, in either straight or branched chain configuration, with from about 3 to about 12 moles of ethylene oxide per mole of alcohol.

10

7. A composition according to claim 5 wherein said nonionic surfactant comprises from about 8% to about 25% of the composition.

15

8. A composition according to claim 2 wherein said amphoteric surfactant comprises the alkali metal, alkaline earth metal, ammonium or substituted ammonium salts of alkyl amphocarboxy glycinate, alkyl amphocarboxypropionate, alkyl amphodipropionate, alkyl amphodiacetate, alkyl amphoglycinate and alkyl amphopropionate wherein alkyl represents an alkyl group having 6 to 20 carbon atoms; alkyl iminopropionate, alkyl iminodipropionate and alkyl amphopropylsulfonate having between 12 and 18 carbon atoms; alkyl betaines and amidopropyl betaines, alkyl sulfonates and alkylamidopropylhydroxy sulfonates wherein alkyl represents an alkyl group having 6 to 20 carbon atoms; and mixtures thereof.

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9. The composition according to claim 8 wherein said amphoteric surfactant is selected from the group consisting of:

a) alkali metal, alkaline earth metal, ammonium or substituted ammonium salts of alkyl amphocarboxy glycinate, alkyl amphocarboxypropionate, alkyl amphodipropionate, alkyl amphodiacetate, alkyl amphoglycinate and alkyl amphopropionate wherein alkyl represents a lauryl or coco group;

30

b) alkyl betaines, amidobetaines, sulfobetaines wherein alkyl represents a lauryl or coco group; and

5 c) mixtures thereof.

10 10. The composition of claim 9 wherein the amphoteric surfactant is selected from the group consisting of cocoamphoacetates, cocoamidopropyl betaines and mixtures thereof.

11. The composition of claim 8 wherein said amphoteric surfactant comprises from about 4% 16% of the composition.

12. A composition according to claim 2 wherein said glycol emulsifier is selected from the group consisting of: propylene glycol, butylene glycol, ethylene glycol and mixtures thereof.

13. A composition according to claim 12 wherein said glycol emulsifier comprises from about 1% to about 10% of the composition.

20 14. A composition according to claim 13 wherein said glycol emulsifier is propylene glycol.

15. A composition according to claim 2 wherein said water comprises from about 25 20% to about 70% of the composition.

16. A cold pearlizing composition consisting essentially of:
a) from about 15% to about 25% of a pearlizing agent selected from the group consisting of ethylene glycol monostearate, ethylene glycol distearate and mixtures thereof;
b) from about 10% to about 20% of a nonionic surfactant selected from the group consisting of condensation products of primary and secondary aliphatic alcohols having from about 8 to about 24 carbon

5 atoms, in either straight or branched chain configuration, with from about 2 to about 40 moles of ethylene oxide per mole of alcohol; condensation products of about 6 to about 12 carbon atom alkyl phenols with about 3 to about 30 moles of ethylene oxide; and mixtures thereof;

10 c) from about 6% to about 10% of an amphoteric surfactant selected from the group consisting of cocoamphoacetates, cocoamidopropyl betaines and mixtures thereof;

d) from about 2% to about 5% of propylene glycol; and

e) from about 20% to about 80% of water.

15 17. A mild shampoo composition comprising the cold pearlizing concentrate of claim 2.

18. A mild hand and body soap composition comprising the cold pearlizing 20 concentrate of claim 2.

19. An ultra-mild bubble bath composition comprising the cold pearlizing concentrate of claim 2.

25 20. A method for preparing an ultra-mild pearlizing concentrate comprising: intimately blending the ingredients of claim 2 at from about 60°C to about 80°C until the pearlizing agent is solubilized.

INTERNATIONAL SEARCH REPORT

International Application No
PCT/US 96/16296

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 A61K7/50 C11D1/94

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 A61K C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP,A,0 376 083 (HENKEL) 4 July 1990 see the whole document ---	1-20
X	WO,A,92 13512 (HENKEL) 20 August 1992 see the whole document ---	1-20
X	WO,A,94 24248 (HENKEL) 27 October 1994 see the whole document ---	1-4, 7-15, 17-20
X,P	US,A,5 529 721 (SALKA ET AL.) 25 June 1996 see the whole document ---	1-4, 7-15, 17-20
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Date of the actual completion of the international search

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